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EFFECT OF SAMPLE HOLDER
GEOMETRY ON PRESSURE-TIME
CURVES OF LEAD DIOXIDE-
CUPRIC OXIDE-SILICON STARTER
COMPOSITION: A NEW TEST
FIXTURE DESIGN

By WILLIAM RIPLEY

U. S. NAVAL AMMUNITION DEPOT

CRANE, INDIANA

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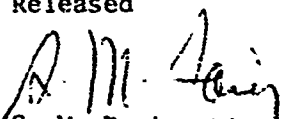
EFFECT OF SAMPLE HOLDER GEOMETRY ON
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OXIDE-SILICON STARTER COMPOSITION:
A NEW TEST FIXTURE DESIGN

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ABSTRACT

Difficulties in interpreting pressure-time curve data obtained on the present test fixture (Mk 25 Marine Location Marker Starter Composition) has led to the design of a new test fixture with a sample geometry which produces a linear plot. A comparison of results obtained on the two test fixtures is made. An optimum range of burning characteristics for the new test fixture is recommended. Also recommended is a basic starter composition formula with enough flexibility to compensate for performance variations due to variations in the reactivity of the chemical ingredients.

EFFECT OF SAMPLE HOLDER GEOMETRY ON
PRESSURE-TIME CURVES OF LEAD DIOXIDE-CUPRIC OXIDE-SILICON
STARTER COMPOSITION: A NEW TEST FIXTURE DESIGN

I. Background

a. A study of the burning characteristics of the lead dioxide-cupric oxide-silicon starter composition used in the Mk 25 Marine Location Marker was published in March 1964.¹ The study utilized pressure-time curves to determine the burning rate--as well as other burning characteristics--of a family of starter mix compositions in which the ratio of the three components were systematically varied. During this study it was also demonstrated that variations in the components themselves could lead to significant variations in the performance of the starter composition.

b. Continuing difficulties with the performance of the starter composition led to a study of the relationship between the properties and the reactivity of lead dioxide specimens obtained from various suppliers. The results of this study, published as RDTR No. 114 in June of 1968², confirmed large variations in the reactivity of the various lead dioxide specimens, variations which also affected the performance of the starter composition. When no clear-cut correlations could be found between various chemical and physical properties of the lead dioxide specimens and the performance of the specimens in starter compositions as indicated by pressure-time curves, it was decided to re-examine the pressure-time curve test. This decision was also based on the experience that the significance of pressure-time curve data is often difficult to

assess, that the extraction of the data is somewhat arbitrary, and that the relationship between different properties of the pressure-time curve--i.e., the slope of the curve, the maximum pressure, and the time to the peak--is not well-enough understood to be mathematically interpreted.

II. Variations in the Pressure-time Curve

a. Obviously, the shape of the pressure-time curve is determined by many factors. It is felt that fundamentally, in terms of burning characteristics, the pressure-time curve depends on the rate of heat evolution and heat loss and the rate of gas evolution and condensation. However, the shape of the pressure-time curve also depends upon experimental factors such as the geometrical shape of the sample holder.

b. Originally, the size and shape of the sample holder was based on the size and shape of the phenolic cup which held the starter composition in the Mk 25 Marine Location Marker: i.e., a cylindrical cavity 0.875 inches in height by 1.50 inches in diameter. The objective was, of course, to make the test condition a facsimile of the actual condition, although aluminum was used instead of plastic. The problem with this choice of geometry of the burning charge is that it involves a constantly changing flame-front and consequently generates a complex curve which is difficult to interpret. For the purposes of analysis and comparison a flame front which quickly reaches steady-state and proceeds accordingly would generate ideally a linear slope. Variations from

this linear slope due to non-homogeneity of the sample or uneven packing could be easily interpreted and corrected by reblending or retesting; whereas, basic differences in the burning characteristics of the composition would show approximate linearity but a different slope. Thus, a sample holder such as Figure 1 is similar in geometry to the actual plastic holder in the signal, but it is difficult to interpret the significance of variations in the pressure-time curve; while a sample holder such as Figure 2 is not similar in geometry to the actual plastic holder in the signal; it furnishes a pressure-time curve that is easy to interpret and to compare with other pressure-time curves. From this standpoint, it is preferable.

c. The difficulty of interpreting variations in pressure-time curves is indicated by Figures 3a and 3b. These are pressure-time curves obtained successively on the same Mk 25 starter composition. Similarly, Figures 4a and 4b show pressure-time curves of another starter composition. Both sets of figures were obtained on the present test fixture, Figure 5, which is located in the Quality Evaluation Laboratory in Building 2044. The significance and cause of such variations are often impossible to explain under prevailing conditions. Do they indicate differences in the starter mix or differences in the experimental conditions? We are not certain.

III. Reproducibility of Results

a. Besides interpretability, reproducibility of results is another important factor in evaluating the significance of pressure-time curves. Consequently, in December of 1968 and January of 1969 a series of tests were performed in the Quality Evaluation Laboratory to evaluate the reproducibility of results obtained on the present test fixture. The results of these tests are shown in Table I. Samples of 2-3-5 starter composition were prepared using 4.3 μ silicon from Hummel Chemical Company, 6.0 μ cupric oxide from Glidden, and lead dioxide from four suppliers. Three series of tests were run on each of the samples. The tests were about two weeks apart. Performance characteristics for 2-3-5 starter composition made from seven lead dioxide samples from five suppliers are shown in Table II.

IV. Design of a New Test Fixture

a. During the last weeks of 1968 a new pressure test fixture was designed and fabricated, Figures 6-8. The essential differences in the new test fixture are the shape and size of the vessel cavity, and the sample holder. The sample holder is a nickel boat (Fisher 7-647) 3" long by 0.75" wide by 0.50" deep which weighs approximately 28 grams. The volume of the vessel is approximately 118 ml. The sample holder is loaded with 15g of 2-3-5 starter composition and ignited with a hot wire which is attached to the screw that connects the sample holder to the lid of the bomb so that ignition conditions are highly reproducible.

Performance characteristics for 2-3-5 starter compositions made from the seven lead dioxide specimens under investigation are shown in Table III for the new test fixture. Reproducibility of performance characteristics of four of these specimens is shown in Table IV.

V. Discussion of Results

a. Data obtained on reproducibility of results shown in Tables I and IV for the present and the new test fixtures are statistically evaluated. The ratio of the pooled variances (the F test) is shown in Table V for the peak pressure, time to Pmax, angle of slope, and tan of the slope. Since $F_{.01}(40, 40) = 2.11$, the F values are significant for each of these characteristics. In general, the data shows that results for the peak pressure is slightly more reproducible for the present test fixture, while the time to Pmax, angle of slope, and tangent of slope are considerably more reproducible for the new test fixture.

b. Difficulties of interpreting pressure-time curves obtained on the present test fixture have already been suggested. Again, these difficulties arise from the condition that a constant changing flame front generates a complex curve which is difficult to interpret and which involves the subjective judgment of the technician (e.g., in drawing the tangent to the slope of the curve). The sample geometry of the new test fixture, on the other hand, allows the burning front through the sample to immediately come to steady-state, so that basically the resulting pressure-time curve

is a linear function. Pressure-time curves for 2-3-5 starter composition made with seven lead dioxide specimens under study are shown in Figures 9a-9g for the new test fixture and in Figures 10a-10g for the present test fixture. Conditions which cause non-linear burning, such as uneven packing or non-homogeneity of the sample, can be readily observed in pressure-time curves obtained on the new test fixture. With the complex curve obtained on the present test fixture, such distinctions are not readily drawn. Nor can it be determined whether or not ignition has occurred in the center of the sample.

c. Generally, the data from both test fixtures are in agreement on the burning rates of the various starter compositions studied, Table VI. In either case, starter composition made with Baker lead dioxide has the fastest burning rate and compositions made from Allied lead dioxide and Eagle-Picher 0.32 μ lead dioxide have the slowest burning rates. All the others fall in about the same order between these extremes. In burning times, Baker composition is the shortest on the present test fixture while Pepcon and Baker are the shortest on the new test fixture. Eagle-Picher 0.32 μ and Allied are the longest on both test fixtures. Pressures obtained on both fixtures are comparable. There is of course less dramatic difference in the peak pressure for the new test fixture because the burning times are slower due to the geometry of the sample. However, this effect which influences peak pressure in the present test fixture influences the slope in

the new test fixtures. Unusual effects such as the presence of gas producing impurities in the silicon are equally obvious using either test fixture, as can be seen in Figures 11 and 12.

d. In the original work done on the present test fixture in 1963, burning characteristics limits were proposed as follows:

- (1) Burning angle, θ $55^\circ - 68^\circ$
- (2) Maximum pressure, P_{max} $6.75 - 9.65$ psi
- (3) Reaction time, t_{Pmax} $6.1 - 7.9$ secs

These values were empirically derived from experience based on the burning characteristics of a larger number of different samples over many months. However, as time passed variations in performance of the composition brought about a relaxation of the above requirements, particularly on the high side. For example, the results in Table VI obtained on the present bomb show five of the seven angles above 68° . Two of the P_{max} values are above the original requirements and all of the burning times are too fast. In general, these compositions appear to be much more reactive than were the typical 1963 compositions shown in Table XV of RDTR No. 41, from which the original requirements were derived. The reasons for this change are not precisely known but it is the rather usual sort of thing that happens to pyrotechnic compositions.

e. The following range of burning characteristics for pressure-time curves obtained on the new test fixture are based on the burning characteristics required on the present test fixture:

- | | |
|---------------------------------|--------------|
| (1) burning rate, θ | 45 - 56° |
| (2) Maximum Pressure, P_{max} | 9.5 - 12 psi |
| (3) Burning time, t_{Pmax} | 10 - 16 secs |

f. However, the real problem is not selecting some arbitrary range for the optimum performance characteristics of the pressure-time curves. Experience ultimately dictates this. The real problem is continual variations in the performance of the compositions due to variations in the properties and behavior of the ingredients used. Present specifications cannot assure ingredients such as lead dioxide or silicon that have consistent and reproducible (predictable) burning properties. If this is true, then what is needed is to establish optimum performance requirements based on the pressure-time curve and to establish a formula range sufficiently broad to enable the performance requirements to be met even using ingredients which have varying reactivities.

VI. Conclusion

a. Arguments have been presented for adopting a pressure test fixture which utilizes a sample geometry that burns at a steady state and develops a linear slope:

1.. Samples with a large cylindrical geometry burn with an increasing and then decreasing flame front that generates a complex pressure-time curve. Such a curve is difficult to interpret and the relationship between different properties of the curve such as peak pressure, slope, and time to peak is obscure. Moreover, values for these properties are often difficult to ascertain.

involve degrees of subjective judgment. On the other hand, a sample geometry that burns to generate a basically linear slope is simple and easy to interpret. Factors such as poor mixing or uneven packing cause non-linearity and can easily be distinguished.

2. Values for the properties of the linear slope are easier to measure, less subjective, and more reproducible.

3. The linear pressure-time curve can be directly compared as to burning rate with burning in the open which can be visually observed and timed. This can be used as a check on instrumentation and a verification of unusual performance.

b. Furthermore, it is recommended that the optimum range of burning characteristics obtained on the new test fixture should be as follows (for 2-3-5 starter composition):

- | | |
|--------------------------------|--------------|
| 1. Burning rate, \dot{e} | 45-55° |
| 2. Maximum Pressure, P_{max} | 9.5-12.5 psi |
| 3. Burning time, t_{Pmax} | 10-16 secs |

c. It is also recommended that a flexible formula such as the following be adopted for the 2-3-5 starter composition:

| Ingredient | % by Weight |
|--------------|---------------|
| Lead dioxide | 20% \pm 10% |
| Cupric oxide | 30% \pm 10% |
| Silicon | 50% \pm 15% |

d. The effects of formula changes on the performance characteristics of this family have already been reported in RDTR No. 41. According to these findings the relationship between the formula and the burning characteristics in a typical composition

can be seen in Table VII. Changes can be of two types. Either the oxidizer-fuel ratio can be held constant and the ratio of lead dioxide-cupric oxide varied, or the ratio of lead dioxide to cupric oxide can be held constant and the fuel-oxidizer ratio varied. It may be noted in Table VII that the 10-40-50 mix has almost exactly the same burning characteristics as the 16-24-60 mix.

e. This data indicates that we can slow down the burning characteristics of a 2-3-5 starter mix by either increasing the silicon content, which decreases the calorific output, or by increasing the cupric oxide-lead dioxide ratio, which increases the calorific output, or by a combination of both changes. Data from Table VI on the starter mix made with Baker lead dioxide indicates that the composition is burning too fast, with an angle of 84.7° and a burning time of 2.5 sec. How can we slow this composition down so that it fits our burning requirements without significantly decreasing the heat of reaction? An increase in silicon will slow it down and decrease its calorific value, and an increase in the cupric oxide-lead dioxide ratio will slow it down but increase the calorific value. Thus, a formula consisting of 17% lead dioxide, 38% cupric oxide, and 55% silicon should burn slower at a lower maximum pressure and yet the heat of reaction should remain about the same, i.e. 352.1 cal/g theoretical. Similarly, data from Table VI on the starter mix made with Eagle-Picher Grade 95 lead dioxide indicates that the composition is burning too slowly, with an angle of 41° and a burning time of

10.5 secs. Here we wish to increase the burning rate without significantly changing the calorific value or the maximum pressure. A formula consisting of 30% lead dioxide, 20% cupric oxide, and 50% silicon should burn more rapidly, with a slight decrease in calorific value and no significant increase in the maximum pressure. Actually, these new formulas were burned in the present test fixture. The starter mix made with Baker lead dioxide had a θ angle of 75° , a maximum pressure of 8.8 psi, and a burn time of 5.35 seconds. The starter mix made with Eagle-Picher Grade 95 lead dioxide had a θ angle of 76.5° , a maximum pressure of 8.4 psi, and a burn time of 4.8 seconds. On the new test fixture the Baker mix had a θ of 50.4° , a pressure of 10.4 psi, and a time of 13.1 seconds. The Eagle-Picher 95 mix had a θ of 54.5° , a Pmax of 10.4 psi, and a burn time of 11.3 seconds. Results on the present test fixture, while similar to the other mixes tested, are still slightly higher than the original specification requirements. A new Baker blend of 15-28-57 gave a θ value of 46° , Pmax 10.5 psi, and time 15 seconds. A new Eagle-Picher 95 blend of 25-20-55 gave a θ value of 47° , Pmax 10.7 psi, and burn time 15.0 seconds.

VII. Future Plans

a. While trends are shown in Table VII which enable one to alter the oxidizer-fuel ratio or the lead dioxide-cupric oxide ratio so as to correct the burning characteristics of a 2-3-5 starter composition which does not meet the specification requirements, further study of these phenomena should yield a

more precise method of arriving at the proper formula change.

b. Study of the 6-6-8 starter composition will be initiated with an objective of determining optimum burning characteristics and procedures for altering the basic formula in order to correct the burning characteristics of compositions that fall outside the established requirements.

LITERATURE CITED

1. Ripley, William, RDTR No. 41, Investigation of the Burning Characteristics of the Lead Dioxide-Cupric Oxide-Silicon Starter Composition, U. S. Naval Ammunition Depot, Crane, Indiana, 26 March 1964.
2. Ripley, William, and Lipscomb, Charles, RDTR No. 114, A Preliminary Investigation of the Reactivity of Lead Dioxide, U. S. Naval Ammunition Depot, Crane, Indiana, 13 June 1968.

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APPENDIX I

TABLE I. Reproducibility of Performance Characteristics Based on Present Test Fixture of 2-3-5 Starter Compositions Made With 4.3 μ Silicon (Hummel), 6.0 μ Cupric Oxide (Glidden), and Lead Dioxide From Four Suppliers.

A. Lead Dioxide from J. T. Baker, Technical Powder, Medium Cure 2352

| | PEAK PRESSURE Pmax, Psi | TIME TO Pmax, Secs | ANGLE OF SLOPE, ° | TAN OF SLOPE |
|-----------|----------------------------|-----------------------|----------------------|-----------------|
| Series 1 | | | | |
| Run #1 | 11.8 | 2.0 | 84 | 9.59 |
| Run #2 | 10.3 | 2.6 | 79 | 5.15 |
| Run #3 | 10.8 | 2.1 | 84 | 9.59 |
| Run #4 | 11.1 | 2.6 | 82 | 7.13 |
| Run #5 | 10.4 | 3.2 | 81.5 | 6.69 |
| Series 2 | | | | |
| Run #1 | 11 | 2.8 | 80 | 5.67 |
| Run #2 | 11 | 2.4 | 82 | 7.13 |
| Run #3 | 10.4 | 2.6 | 81 | 6.32 |
| Series 3 | | | | |
| Run #1 | 10.5 | 4.8 | 82 | 7.13 |
| Run #2 | 10.6 | 2.8 | 84 | 9.59 |
| Run #3 | 10.6 | 4.3 | 78 | 4.71 |
| \bar{X} | 10.77 | 2.93 | 81.59 | 7.15 |
| σ | .44 | .87 | 2.01 | 1.76 |

B. Lead Dioxide from Eagle-Picher 0.52, Pack 2-w-294 G/O #00290

| | PEAK PRESSURE Pmax, Psi | TIME TO Pmax, Secs | ANGLE OF SLOPE, ° | TAN OF SLOPE |
|-----------|----------------------------|-----------------------|----------------------|-----------------|
| Series 1 | | | | |
| Run #1 | 9.85 | 3.2 | 75.5° | 3.87 |
| Run #2 | 9.45 | 3.5 | 74.5 | 3.60 |
| Run #3 | 9.95 | 3.0 | 77.5 | 4.53 |
| Run #4 | 9.7 | 3.8 | 77.0 | 4.34 |
| Run #5 | 9.8 | 4.4 | 75.0 | 3.74 |
| Series 2 | | | | |
| Run #1 | 9.5 | 3.2 | 78.0 | 4.71 |
| Run #2 | 10.1 | 3.1 | 77.0 | 4.34 |
| Run #3 | 9.5 | 3.9 | 77.0 | 4.34 |
| Series 3 | | | | |
| Run #1 | 10.5 | 3.4 | 70 | 2.74 |
| Run #2 | 10.25 | 2.6 | 77 | 4.34 |
| Run #3 | 10.0 | 5.8 | 78 | 4.71 |
| \bar{X} | 9.87 | 3.63 | 76.05 | 4.11 |
| σ | .11 | .87 | 2.32 | .59 |

C. Lead Dioxide from Pepcon, Pacific Engineering and Production Company

| | PEAK PRESSURE Pmax, Psi | TIME TO Pmax, Secs | ANGLE OF SLOPE, ° | TAN OF SLOPE |
|-----------|----------------------------|-----------------------|----------------------|-----------------|
| Series 1 | | | | |
| Run #1 | 7.95 | 4.0 | 74 | 3.48 |
| Run #2 | 8.15 | 3.4 | 72 | 3.08 |
| Run #3 | 8.2 | 3.0 | 77 | 4.34 |
| Run #4 | 8.15 | 3.8 | 78.5 | 4.92 |
| Run #5 | 8.2 | 2.8 | 75.5 | 3.87 |
| Series 2 | | | | |
| Run #1 | 8.2 | 2.6 | 75 | 3.74 |
| Run #2 | 8.2 | 2.5 | 77 | 4.34 |
| Run #3 | 8.3 | 3.9 | 75 | 3.74 |
| Series 3 | | | | |
| Run #1 | 8.75 | 2.5 | 78 | 4.71 |
| Run #2 | 8.25 | 4.5 | 74 | 3.48 |
| Run #3 | 8.5 | 3.2 | 74 | 3.48 |
| \bar{X} | 8.26 | 3.29 | 75.45 | 3.93 |
| σ | .21 | .68 | 1.98 | .58 |

D. Lead Dioxide from Shepherd Chemical Company, Dense, Government Specification, Lot 5337

| | PEAK PRESSURE Pmax, Psi | TIME TO Pmax, Secs | ANGLE OF SLOPE, ° | TAN OF SLOPE |
|-----------|----------------------------|-----------------------|----------------------|-----------------|
| Series 1 | | | | |
| Run #1 | 7.8 | 5.5 | 64 | 2.05 |
| Run #2 | 8.1 | 3.2 | 75 | 3.74 |
| Run #3 | 8.15 | 4.0 | 69 | 2.68 |
| Run #4 | 8.3 | 3.5 | 72 | 3.08 |
| Run #5 | 8.1 | 3.2 | 78 | 4.71 |
| Series 2 | | | | |
| Run #1 | 8.3 | 3.2 | 72 | 3.08 |
| Run #2 | 8.0 | 6.4 | 70 | 2.74 |
| Run #3 | 8.1 | 3.0 | 73 | 3.27 |
| Series 3 | | | | |
| Run #1 | 7.25 | 6.4 | 59 | 1.66 |
| Run #2 | 7.25 | 6.0 | 73 | 3.27 |
| Run #3 | 8.25 | 5.5 | 75 | 3.74 |
| \bar{X} | 7.96 | 4.55 | 70.91 | 3.09 |
| σ | .38 | 1.42 | 5.38 | .83 |

TABLE II. Performance Characteristics Based on Present Test Fixture of 2-3-5 Starter Composition Made With 4.3 μ Silicon (Hummel), 6.0 μ Cupric Oxide (Glidden), and Lead Dioxide from Various Suppliers.

A. Lead Dioxide from Allied Chemical, B&A, ACS Reagent Powder, Code 1843

| | PEAK PRESSURE Pmax, Fsi | TIME TO Pmax, Secs | ANGLE OF SLOPE, ° | TAN OF SLOPE |
|-----------|----------------------------|-----------------------|----------------------|-----------------|
| Run #1 | 8.1 | 4.6 | 66.5 | 2.29 |
| Run #2 | 8.1 | 6.1 | 52.5 | 1.32 |
| Run #3 | 7.9 | 6.1 | 55.5 | 1.45 |
| Run #4 | 8.2 | 5.9 | 56.5 | 1.59 |
| Run #5 | 8.1 | 4.9 | 67.0 | 2.35 |
| \bar{X} | 8.08 | 5.52 | 59.60 | 1.80 |
| σ | .11 | .72 | 6.67 | .48 |

B. Lead Dioxide from J. T. Baker, Technical Powder, Medium Cure, 2352

| | | | | |
|-----------|-------|------|-------|------|
| Run #1 | 11.8 | 2.0 | 84.0 | 9.59 |
| Run #2 | 10.3 | 2.6 | 79.0 | 5.15 |
| Run #3 | 10.8 | 2.1 | 84.0 | 9.59 |
| Run #4 | 11.1 | 2.6 | 87.0 | 7.13 |
| Run #5 | 10.4 | 3.2 | 89.5 | 6.69 |
| \bar{X} | 10.88 | 2.50 | 84.70 | 7.63 |
| σ | .61 | .48 | 3.93 | 1.93 |

C. Lead Dioxide from Eagle-Picher 0.32 Pack 2-w-378 G/O #00290

| | | | | |
|-----------|------|------|-------|--|
| Run #1 | 9.1 | 5.5 | 65.5 | |
| Run #2 | 9.6 | 5.1 | 65.0 | |
| Run #3 | 9.9 | 6.8 | 58.5 | |
| Run #4 | 9.3 | 7.5 | 57.0 | |
| Run #5 | 9.65 | 5.4 | 63.0 | |
| \bar{X} | 9.51 | 6.06 | 61.80 | |
| σ | .31 | 1.04 | 3.85 | |

L. Lead Dioxide from Eagle-Picher 0.52 Pack #2-w-394 G/O #00290

| | PEAK PRESSURE Pmax, Psi | TIME TO Pmax, Secs | ANGLE OF SLOPE, ° | TAN OF SLOPE |
|-----------|----------------------------|-----------------------|----------------------|-----------------|
| Run #1 | 9.1 | 5.5 | 65.5 | |
| Run #2 | 9.6 | 5.1 | 65.0 | |
| Run #3 | 9.9 | 6.8 | 58.5 | |
| Run #4 | 9.3 | 7.5 | 57.0 | |
| Run #5 | 9.65 | 5.4 | 63.0 | |
| \bar{X} | 9.51 | 6.06 | 61.80 | |
| σ | .31 | 1.04 | 3.85 | |

E. Lead Dioxide from Eagle-Picher .66 Pack #2-w-286 G/O #00290

| | | | | |
|-----------|------|------|------|--|
| Run #1 | 8.9 | 3.0 | 76 | |
| Run #2 | 7.97 | 2.8 | 78.5 | |
| Run #3 | 9.1 | 4.0 | 76 | |
| Run #4 | 9.7 | 2.8 | 80 | |
| Run #5 | 9.75 | 3.4 | 73 | |
| \bar{X} | 9.08 | 3.20 | 76.7 | |
| σ | .72 | .51 | 2.68 | |

F. Lead Dioxide from Pacific Engineering Production Company

| | | | | |
|-----------|------|-----|-------|--|
| Run #1 | 7.95 | 4.0 | 74 | |
| Run #2 | 8.15 | 3.4 | 72 | |
| Run #3 | 8.2 | 3.0 | 77 | |
| Run #4 | 8.15 | 3.8 | 78.5 | |
| Run #5 | 8.2 | 2.8 | 75.5 | |
| \bar{X} | 8.13 | 3.4 | 75.40 | |
| σ | .10 | .51 | 2.53 | |

G. Shepherd Chemical Company, Dense, Government Specification
Lot 5337

| | PEAK PRESSURE Pmax, Psi | TIME TO Pmax, Secs | ANGLE OF SLOPE, ° | TAN OF SLOPE |
|-----------|----------------------------|-----------------------|----------------------|-----------------|
| Run #1 | 7.8 | 5.6 | 64 | |
| Run #2 | 8.1 | 3.2 | 75 | |
| Run #3 | 8.15 | 4.0 | 69 | |
| Run #4 | 8.3 | 3.5 | 72 | |
| Run #5 | 8.1 | 3.2 | 78 | |
| \bar{X} | 8.09 | 3.90 | 71.60 | |
| σ | .18 | 1.005 | 5.41 | |

TABLE III. Performance Characteristics Based on New Design Test Fixture of 2-3-5 Starter Composition Made with 4.3 μ Silicon (Hummel), 6.0 μ Cupric Oxide (Glidden), and Lead Dioxide from Various Suppliers.

A. Lead Dioxide from Allied Chemical, B&A, ACS Reagent Powder, Code 1843

| | PEAK PRESSURE Pmax, Psi | TIME TO Pmax, Secs | ANGLE OF SLOPE, ° | TAN OF SLOPE | OPEN BURNING RATE Secs/3inches |
|-----------|----------------------------|-----------------------|----------------------|-----------------|-----------------------------------|
| Run #1 | 11.0 | 17.2 | 43.7° | 0.96 | 15.8 |
| Run #2 | 11.5 | 15.4 | 48.3 | 1.12 | 15.9 |
| Run #3 | 10.4 | 15.4 | 45.4 | 1.01 | 16.7 |
| Run #4 | 10.6 | 15.4 | 45.9 | 1.03 | 15.9 |
| Run #5 | 10.6 | 15.0 | 46.7 | 1.06 | 16.1 |
| \bar{X} | 10.82 | 15.68 | 45.92 | 1.036 | 16.08 |
| σ | .44 | .87 | 1.71 | .059 | .36 |

B. Lead Dioxide from J. T. Baker, Technical Powder, Medium Cure, 2352

| | | | | | |
|-----------|-------|-------|-------|-------|-------|
| Run #1 | 12.1 | 11.2 | 59.2° | 1.68 | 13.6 |
| Run #2 | 12.5 | 11.2 | 59.0 | 1.67 | 13.7 |
| Run #3 | 12.75 | 11.2 | 59.8 | 1.72 | 13.6 |
| Run #4 | 12.6 | 11.6 | 58.3 | 1.62 | 12.9 |
| Run #5 | 12.6 | 11.2 | 59.2 | 1.68 | 13.5 |
| \bar{X} | 12.51 | 11.28 | 59.1 | 1.674 | 13.46 |
| σ | .25 | .18 | .54 | .036 | .32 |

C. Lead Dioxide from Eagle Picher 0.32 Pack #2-w-378 G/O #00290

| | | | | | |
|-----------|-------|-------|-------|------|-------|
| Run #1 | 12.5 | 19.1 | 44.4 | .98 | 21.1 |
| Run #2 | 12.5 | 19.5 | 43.8 | .96 | 20.7 |
| Run #3 | 12.8 | 19.5 | 44.8 | .99 | 20.6 |
| Run #4 | 12.75 | 19.5 | 44.5 | .98 | 22.6 |
| Run #5 | 13.7 | 18.7 | 47.5 | 1.09 | 22.7 |
| \bar{X} | 12.85 | 19.26 | 45.00 | 1.00 | 21.54 |
| σ | .49 | .36 | 1.44 | .05 | 1.03 |

D. Lead Dioxide from Eagle-Picher 0.52 Pack #2-w-394 G/O #00290

| | PEAK PRESSURE Pmax, Psi | TIME TO Pmax, Secs | ANGLE OF SLOPE, ° | TAN OF SLOPE | OPEN BURNING RATE Secs/3 inches |
|-----------|----------------------------|-----------------------|----------------------|-----------------|------------------------------------|
| Run #1 | 12.5 | 19.1 | 44.4 | .98 | 21.1 |
| Run #2 | 12.5 | 19.5 | 43.8 | .96 | 20.7 |
| Run #3 | 12.8 | 19.5 | 44.8 | .99 | 20.6 |
| Run #4 | 12.75 | 19.5 | 44.5 | .98 | 22.6 |
| Run #5 | 13.7 | 18.7 | 47.5 | 1.09 | 22.7 |
| \bar{X} | 12.85 | 19.26 | 45.00 | 1.00 | 21.54 |
| σ | .49 | .36 | 1.44 | .05 | 1.03 |

E. Lead Dioxide from Eagle-Picher 0.66 Pack #2-w-286 G/O #00290

| | | | | | |
|-----------|-------|-------|-------|-------|-------|
| Run #1 | 11.0 | 11.7 | 54.6 | 1.41 | 13.5 |
| Run #2 | 11.5 | 11.9 | 55.4 | 1.45 | 13.2 |
| Run #3 | 11.6 | 11.8 | 55.8 | 1.47 | 13.3 |
| Run #4 | 11.5 | 11.8 | 55.6 | 1.46 | 13.8 |
| Run #5 | 11.0 | 11.6 | 54.8 | 1.42 | 13.7 |
| Run #6 | 11.0 | 11.6 | 52.9 | 1.32 | |
| \bar{X} | 11.27 | 11.73 | 54.85 | 1.428 | 13.50 |
| σ | .29 | .12 | 1.06 | .057 | .25 |

F. Lead Dioxide from Pepcon, Pacific Engineering Production Company

| | | | | | |
|-----------|-------|-------|-------|-------|-------|
| Run #1 | 10.5 | 10.8 | 55.4 | 1.45 | 13.3 |
| Run #2 | 9.9 | 11.3 | 53.0 | 1.33 | 12.5 |
| Run #3 | 11.5 | 10.1 | 59.0 | 1.67 | 13.0 |
| Run #4 | 10.5 | 10.5 | 56.4 | 1.50 | 12.4 |
| Run #5 | 10.75 | 11.1 | 55.6 | 1.46 | 11.5 |
| \bar{X} | 10.63 | 10.76 | 55.88 | 1.482 | 12.54 |
| σ | | | | | |

G. Shepherd Chemical Company, Dense, Government Specification
Lot 5337

| | PEAK PRESSURE Pmax, Psi | TIME TO Pmax, Secs | ANGLE OF SLOPE, ° | TAN OF SLOPE | OPEN BURNING RATE Secs/3 inches |
|-----------|----------------------------|-----------------------|----------------------|-----------------|------------------------------------|
| Run #1 | 9.6 | 14.2 | 46.4° | 1.05 | 15.5 |
| Run #2 | 9.7 | 14.2 | 45.7 | 1.03 | 15.4 |
| Run #3 | 10.1 | 13.5 | 48.2 | 1.12 | 14.5 |
| Run #4 | 9.5 | 13.5 | 46.5 | 1.06 | 15.1 |
| Run #5 | 9.8 | 13.9 | 46.9 | 1.07 | 15.1 |
| \bar{X} | 9.74 | 13.86 | 46.74 | 1.066 | 15.12 |
| σ | .23 | .35 | .92 | .034 | .39 |

TABLE IV. Reproducibility of Performance Characteristics Based on New Design Test Fixture of 2-3-5 Starter Composition Made with 4.3 μ Silicon (Hummel), 6.0 μ Cupric Oxide (Glidden), and Lead Dioxide from Various Suppliers.

A. Lead Dioxide from J. T. Baker, Technical Powder, Medium Cure, 2352.

| | PEAK PRESSURE Pmax, Psi | TIME TO Pmax, Psi | ANGLE OF SLOPE, ° | TAN OF SLOPE | |
|-----------|----------------------------|----------------------|----------------------|-----------------|--|
| Series 1 | | | | | |
| Run #1 | 12.1 | 11.25 | 59.2 | 1.68 | |
| Run #2 | 12.5 | 11.25 | 59.0 | 1.67 | |
| Run #3 | 12.7 | 11.25 | 59.3 | 1.72 | |
| Run #4 | 12.6 | 11.6 | 58.3 | 1.62 | |
| Run #5 | 12.6 | 11.25 | 59.2 | 1.68 | |
| Series 2 | | | | | |
| Run #1 | 11.8 | 12.0 | 56.4 | 1.50 | |
| Run #2 | 11.8 | 11.2 | 57.5 | 1.57 | |
| Run #3 | 11.5 | 11.2 | 56.8 | 1.53 | |
| Series 3 | | | | | |
| Run #1 | 11.4 | 11.8 | 55.4 | 1.45 | |
| Run #2 | 11.3 | 11.6 | 55.4 | 1.45 | |
| Run #3 | 11.7 | 12.0 | 55.4 | 1.45 | |
| \bar{X} | 12.00 | 11.49 | 57.49 | 1.575 | |
| σ | .52 | .32 | 1.70 | .104 | |

B. Lead Dioxide from Eagle-Picher, 0.52 μ Pack #2-w-394 G/O #06290

| | PEAK PRESSURE Pmax, Psi | TIME TO Pmax, Fsi | ANGLE OF SLOPE, ° | TAN OF SLOPE |
|-----------|----------------------------|----------------------|----------------------|-----------------|
| Series 1 | | | | |
| Run #1 | 11.4 | 13.4 | 52.3 | 1.29 |
| Run #2 | 11.5 | 13.15 | 52.7 | 1.31 |
| Run #3 | 11.4 | 13.15 | 52.7 | 1.31 |
| Run #4 | 12.0 | 13.15 | 53.9 | 1.37 |
| Run #5 | 11.3 | 13.15 | 51.9 | 1.28 |
| Series 2 | | | | |
| Run #1 | 11.1 | 14.0 | 49.7 | 1.18 |
| Run #2 | 11.2 | 13.0 | 52.2 | 1.29 |
| Run #3 | 11.4 | 13.7 | 50.9 | 1.23 |
| Series 3 | | | | |
| Run #1 | 11.25 | 13.8 | 51.0 | 1.23 |
| Run #2 | 11.20 | 13.65 | 51.0 | 1.23 |
| Run #3 | 11.50 | 13.2 | 52.4 | 1.30 |
| \bar{X} | 11.386 | 13.40 | 51.88 | 1.27 |
| σ | .241 | .34 | 1.15 | .053 |

C. Lead Dioxide from Pepcon, Pacific Engineering Production Company

| | PEAK PRESSURE Pmax, Psi | TIME TO Pmax, Secs | ANGLE OF SLOPE, ° | TAN OF SLOPE |
|-----------|----------------------------|-----------------------|----------------------|-----------------|
| Series 1 | | | | |
| Run #1 | 10.5 | 10.8 | 55.4° | 1.45 |
| Run #2 | 9.9 | 11.3 | 53.0 | 1.33 |
| Run #3 | 11.5 | 10.1 | 59.0 | 1.67 |
| Run #4 | 10.5 | 10.5 | 56.4 | 1.50 |
| Run #5 | 10.75 | 11.1 | 55.6 | 1.46 |
| Series 2 | | | | |
| Run #1 | 9.2 | 11.1 | 51.1° | 1.24 |
| Run #2 | 9.8 | 10.6 | 54.2 | 1.39 |
| Run #3 | 9.3 | 11.2 | 51.1 | 1.24 |
| Series 3 | | | | |
| Run #1 | 9.4 | 11.4 | 52.7 | 1.31 |
| Run #2 | 9.7 | 10.6 | 53.5 | 1.35 |
| Run #3 | 9.7 | 10.2 | 54.7 | 1.41 |
| \bar{X} | 10.02 | 10.81 | 54.25 | 1.395 |
| σ | .71 | .44 | 2.34 | .125 |

D. Lead Dioxide from Shepherd Chemical Company, Dense,
Government Specification, Lot 5337

| | PEAK PRESSURE Pmax, Psi | TIME TO Pmax, Psi | ANGLE OF SLOPE, ° | TAN OF SLOPE |
|-----------|----------------------------|----------------------|----------------------|-----------------|
| Series 1 | | | | |
| Run #1 | 9.6 | 14.2 | 46.4 | 1.05 |
| Run #2 | 9.2 | 14.2 | 45.7 | 1.03 |
| Run #3 | 10.1 | 13.5 | 48.2 | 1.12 |
| Run #4 | 9.5 | 13.5 | 46.5 | 1.06 |
| Run #5 | 9.8 | 13.9 | 46.9 | 1.07 |
| Series 2 | | | | |
| Run #1 | 9.2 | 13.6 | 45.0 | 1.00 |
| Run #2 | 9.3 | 13.5 | 46.5 | 1.02 |
| Run #3 | 9.0 | 14.0 | 44.0 | 0.96 |
| Series 3 | | | | |
| Run #1 | 9.2 | 13.7 | 45.0 | 1.00 |
| Run #2 | 9.7 | 13.9 | 46.5 | 1.05 |
| Run #3 | 9.8 | 12.8 | 49.3 | 1.16 |
| \bar{X} | 9.49 | 13.71 | 46.28 | 1.047 |
| σ | .34 | .40 | 1.50 | .056 |

TABLE V. Ratio of Square of Standard Deviation Variances on Burning Characteristics of 2-3-5 Starter Composition. Made with Four Specimens of Lead Dioxide

| SAMPLE | PEAK PRESSURE | | TIME TO PEAK | | ANGLE OF SLOPE | | TANGENT OF ANGLE | |
|----------------------------|----------------------|------------------|----------------------|------------------|----------------------|------------------|----------------------|------------------|
| | Present Test Fixture | New Test Fixture | Present Test Fixture | New Test Fixture | Present Test Fixture | New Test Fixture | Present Test Fixture | New Test Fixture |
| Baker | 0.44 | 0.52 | 0.87 | 0.32 | 2.01 | 1.70 | 1.76 | 0.10 |
| Eagle-Picher 0.52 μ | 0.11 | 0.24 | 0.87 | 0.34 | 2.32 | 1.15 | 0.59 | 0.05 |
| Pepcon | 0.21 | 0.71 | 0.68 | 0.44 | 1.98 | 2.34 | 0.58 | 0.13 |
| Shepherd | 0.38 | 0.34 | 1.42 | 0.40 | 5.38 | 1.50 | 0.83 | 0.06 |
| $F_{(40,40)}$ | 2.404 | | 6.985 | | 3.542 | | 138.1 | |

TABLE VI. Comparison of Burning Characteristics of 2-3-5 Starter Composition Made with Eight Different Lead Dioxide Specimens

Results Obtained on Present Test Fixture

| PbO ₂ SOURCE | ANGLE OF SLOPE, ° | PRESSURE P _{max} , Psi | TIME TO P _{max} , Sec |
|----------------------------|----------------------|------------------------------------|-----------------------------------|
| Allied | 59.6° | 8.08 | 5.52 |
| Baker | 84.7 | 10.90 | 2.50 |
| Eagle- Picher 0.32μ | 61.8 | 9.50 | 6.06 |
| Eagle- Picher 0.52μ | 75.9 | 9.75 | 3.58 |
| Eagle- Picher 0.66μ | 76.7 | 9.08 | 3.20 |
| Eagle- Picher 95 | 41 | 7.3 | 10.5 |
| Pepcon | 77.4 | 8.13 | 3.40 |
| Shepherd | 71.6 | 8.09 | 3.90 |

Results Obtained on New Test Fixture

| | | | |
|------------------------|------|------|------|
| Allied | 45.9 | 10.8 | 15.7 |
| Baker | 59.1 | 12.5 | 11.3 |
| Eagle- Picher 0.32μ | 45.0 | 12.8 | 19.3 |
| Eagle- Picher 0.52μ | 52.7 | 11.5 | 13.2 |
| Eagle- Picher 0.66μ | 54.8 | 11.3 | 11.7 |
| Eagle- Picher 95 | 31.5 | 10.4 | 25.5 |
| Pepcon | 55.9 | 10.6 | 10.8 |
| Shepherd | 46.7 | 9.7 | 13.9 |

TABLE VII. Effects of Formula Changes on the Performance Characteristics of the Lead Dioxide--Cupric Oxide--Silicon Family

Oxidizer--Fuel Ratio Constant; Lead Dioxide--Cupric Oxide Varying (Present Test Fixture)

| PbO ₂ | CuO | Silicon | θ | Pmax | tPmax | ΔH | |
|------------------|-----|---------|----------|-------|-------|------------|--|
| 10% | 40% | 50% | 45° | 6.45 | 8.38 | 407.4 | |
| 20 | 30 | 50 | 66 | 7.90 | 7.38 | 384.3 | |
| 30 | 20 | 50 | 74 | 8.38 | 3.25 | 362.0 | |
| 40 | 10 | 50 | 82 | 10.40 | 2.00 | 339.0 | |

Lead Dioxide--Cupric Oxide Ratio Constant; Oxidizer--Fuel Ratio Varying (Present Test Fixture)

| PbO ₂ | CuO | Silicon | θ | Pmax | tPmax | ΔH | |
|------------------|-----|---------|----------|------|-------|------------|--|
| 24% | 36% | 40% | 56° | 9.75 | 7.88 | 461.6 | |
| 20 | 30 | 50 | 66 | 7.90 | 7.38 | 384.3 | |
| 16 | 24 | 60 | 45 | 6.5 | 8.75 | 307.8 | |

Figure 1. Sample Holder Used in Present
Pressure Test Fixture

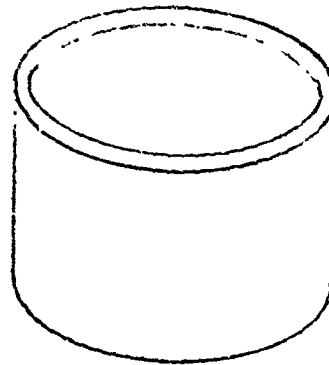


Figure 2. Sample Holder Used in New
Pressure Test Fixture

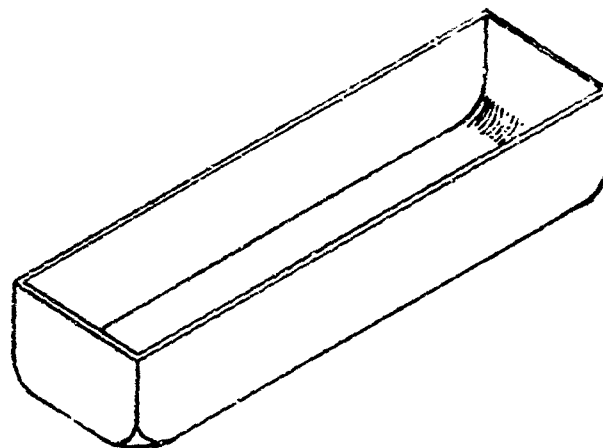
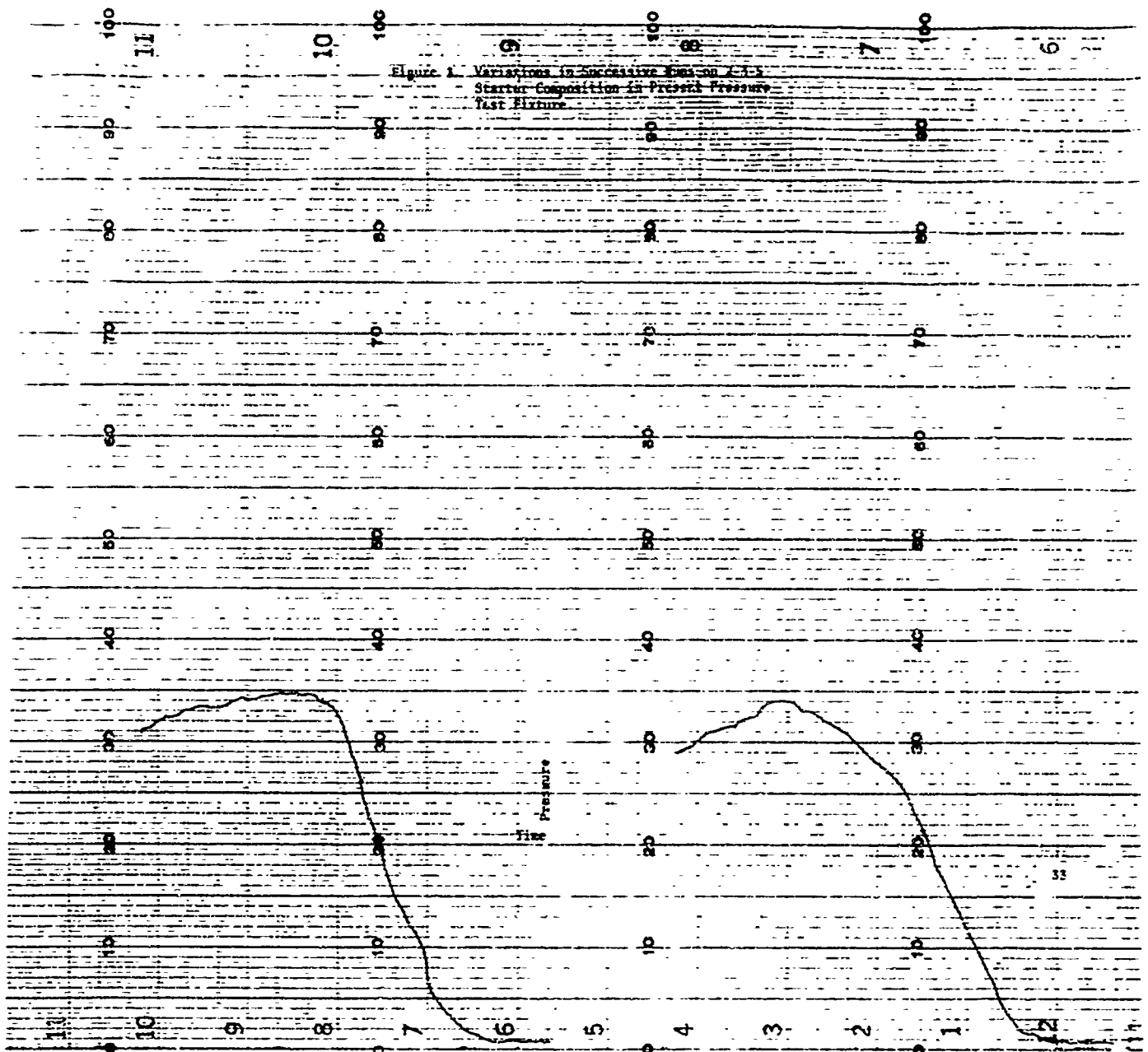
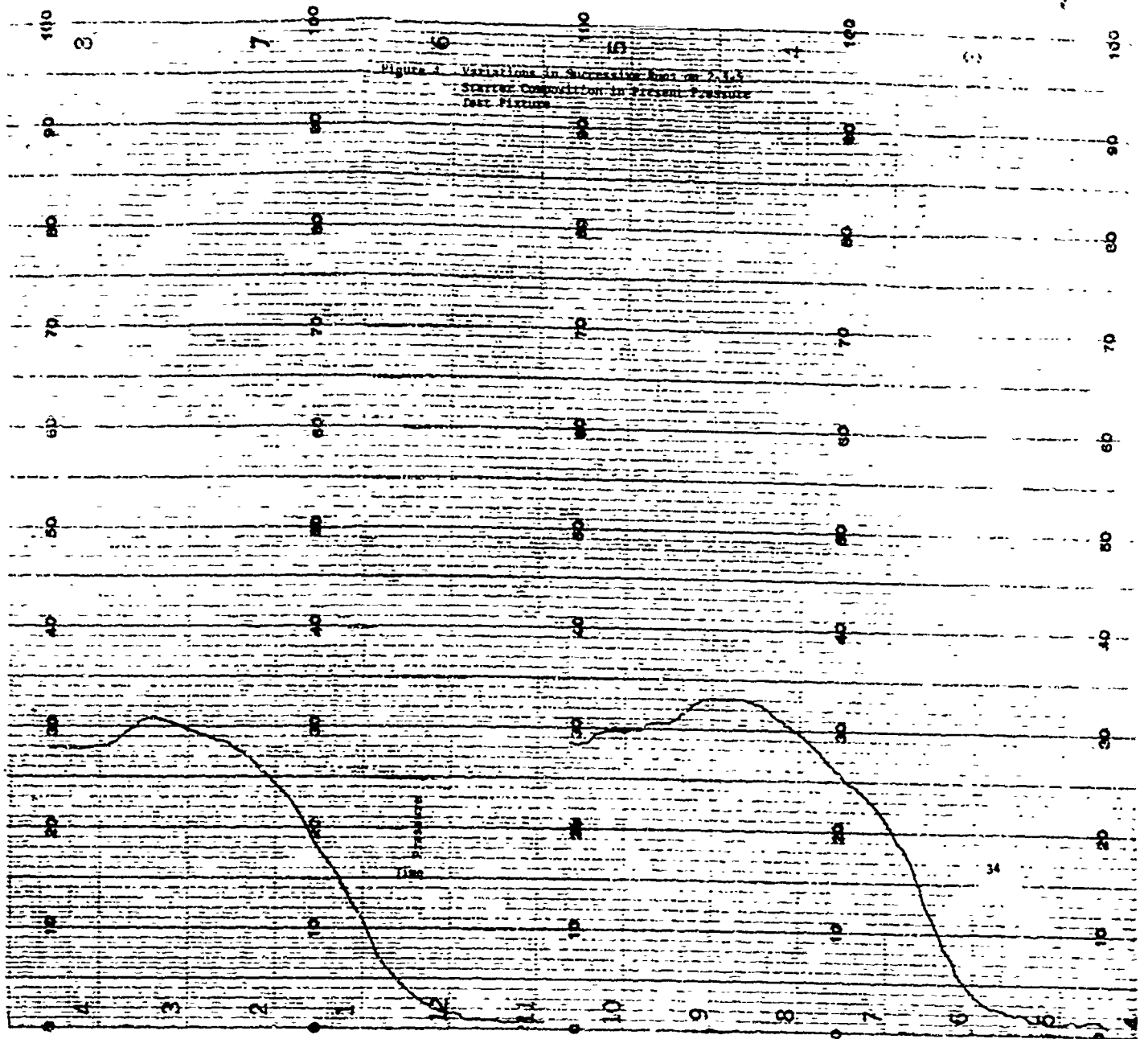
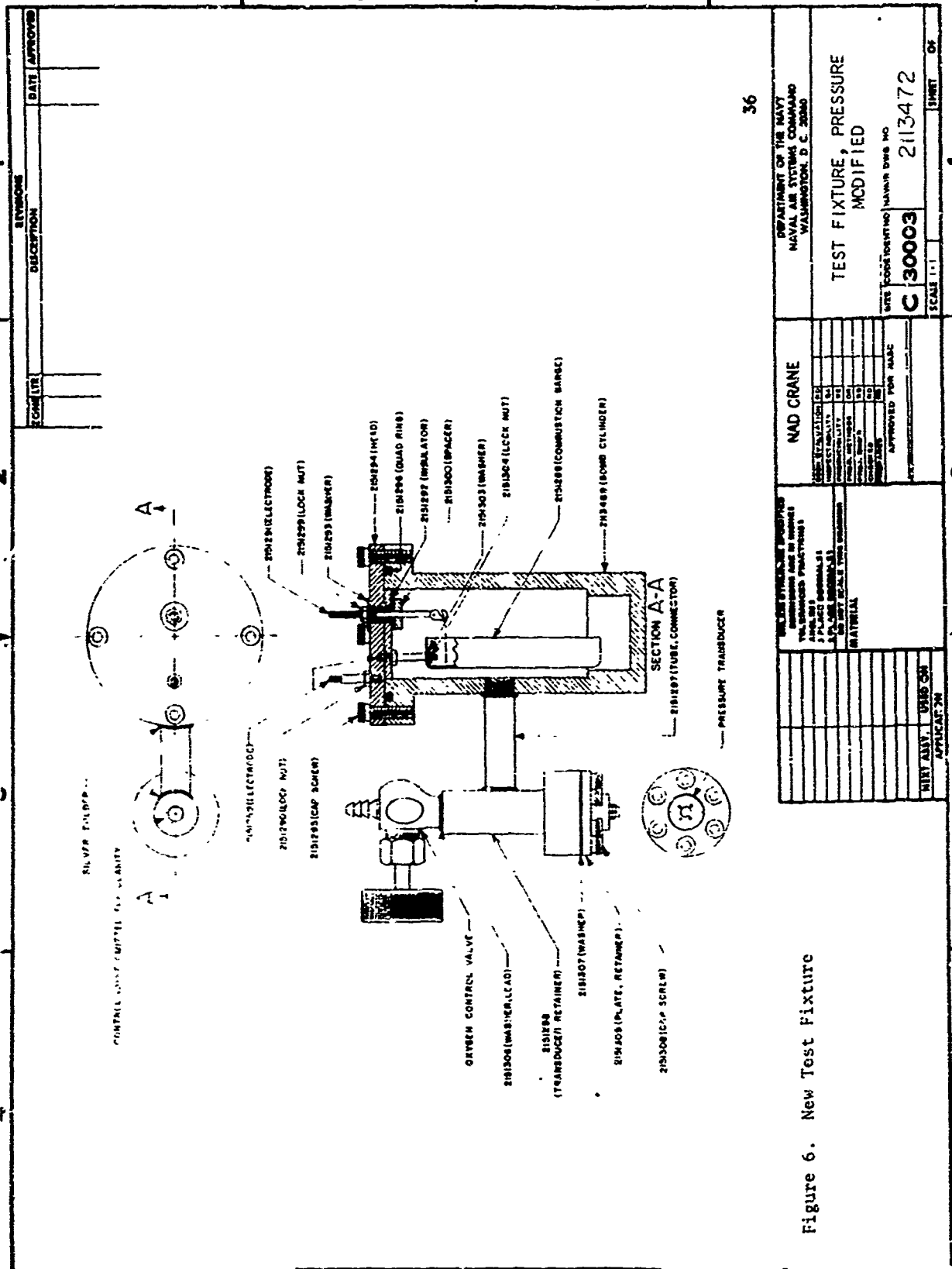


Figure 1. Variations in Successive Runs on C-1-5
 Starter Composition in Pressure
 Test Fixture







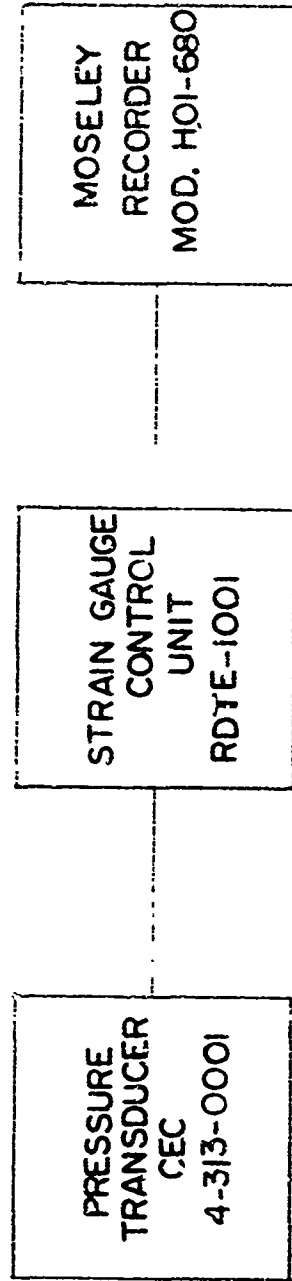


FIGURE 7. ELECTRONIC COMPONENTS FOR NEW PRESSURE
TEST FIXTURE

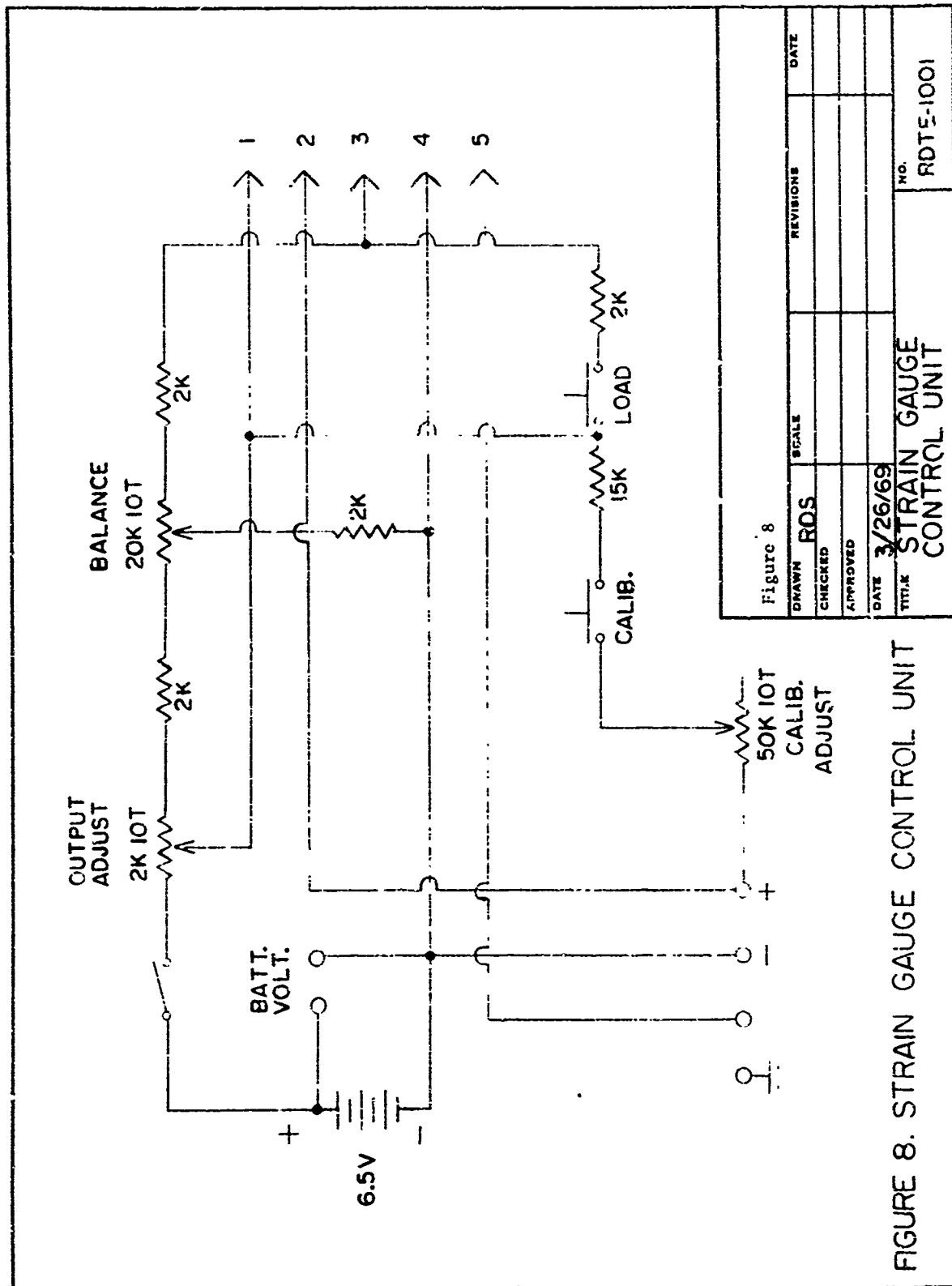
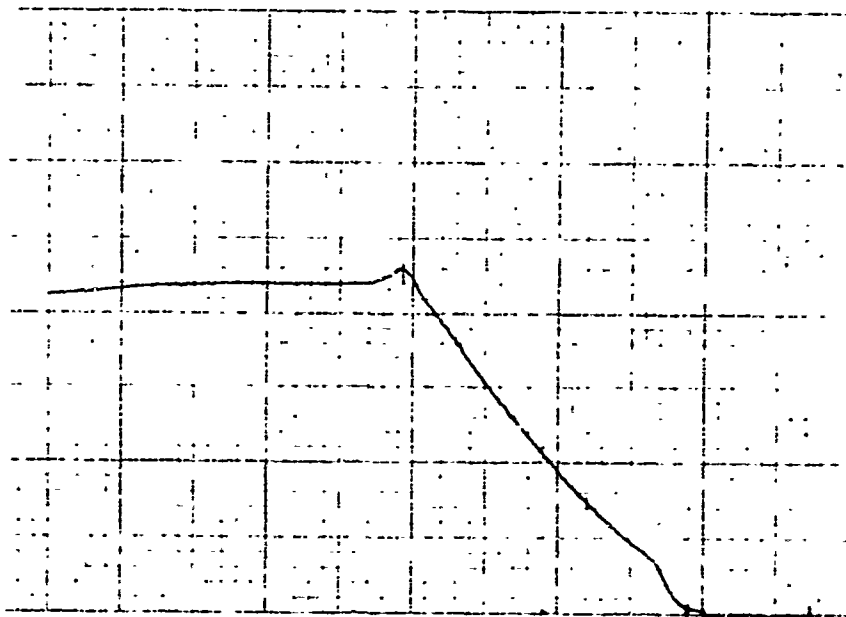
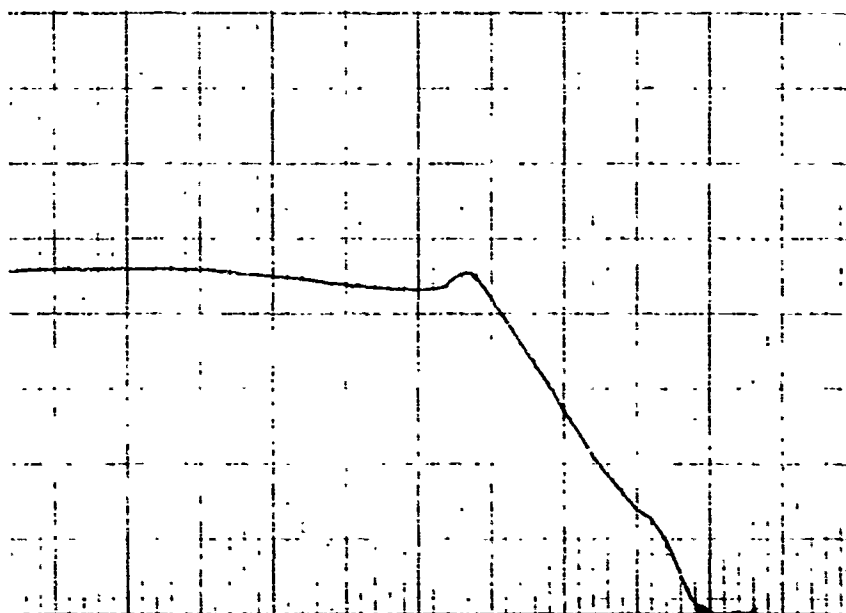


Figure 9. Pressure-time Curves of 2-S-5 Starter Composition Made with Different Lead Dioxide Specimens Using New Test Fixture

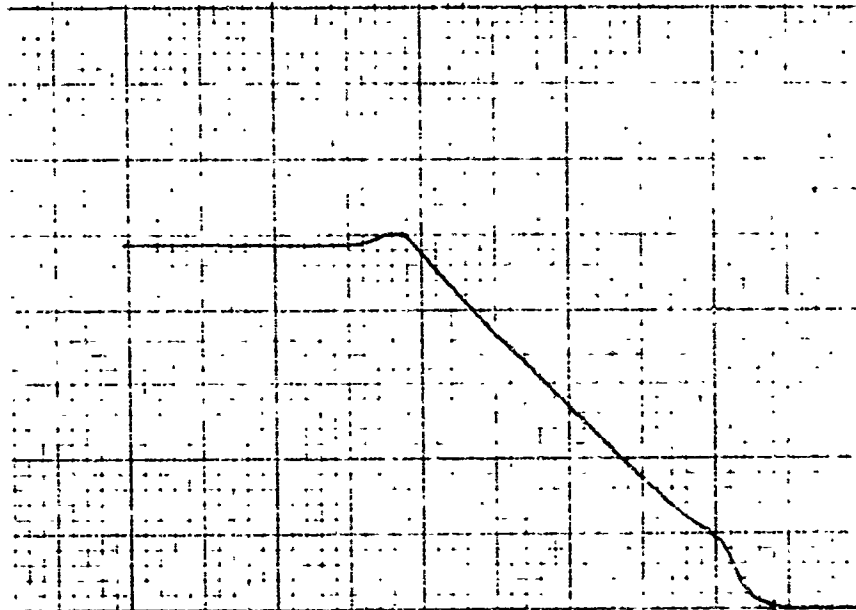
a. Allied Lead Dioxide



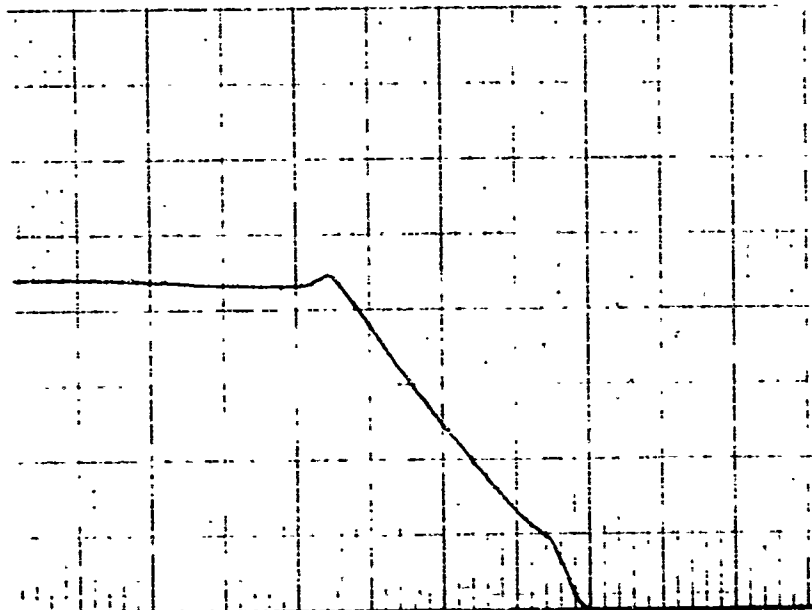
b. Baker Lead Dioxide



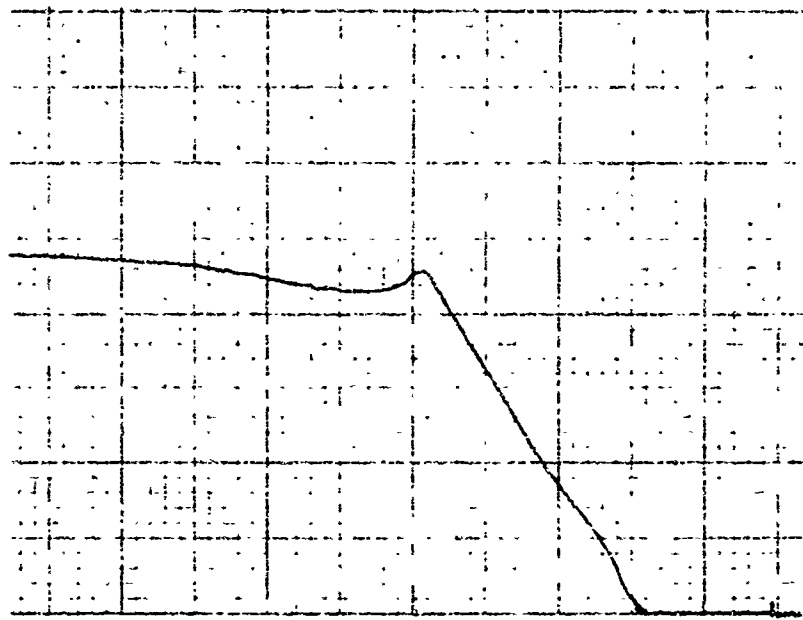
c. Eagle-Picher 0.32 Lead Dioxide



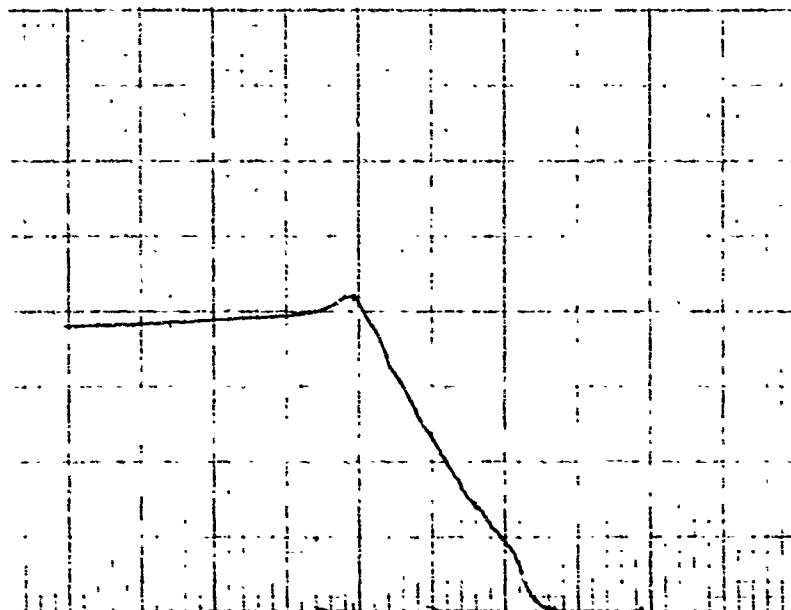
d. Eagle-Picher 0.52 Lead Dioxide



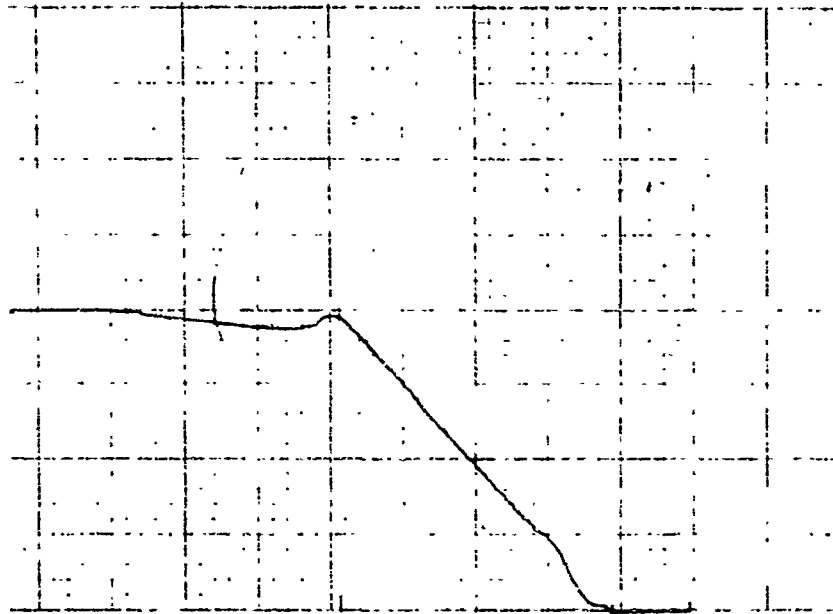
e. Eagle-Picher 0.66 Lead Dioxide

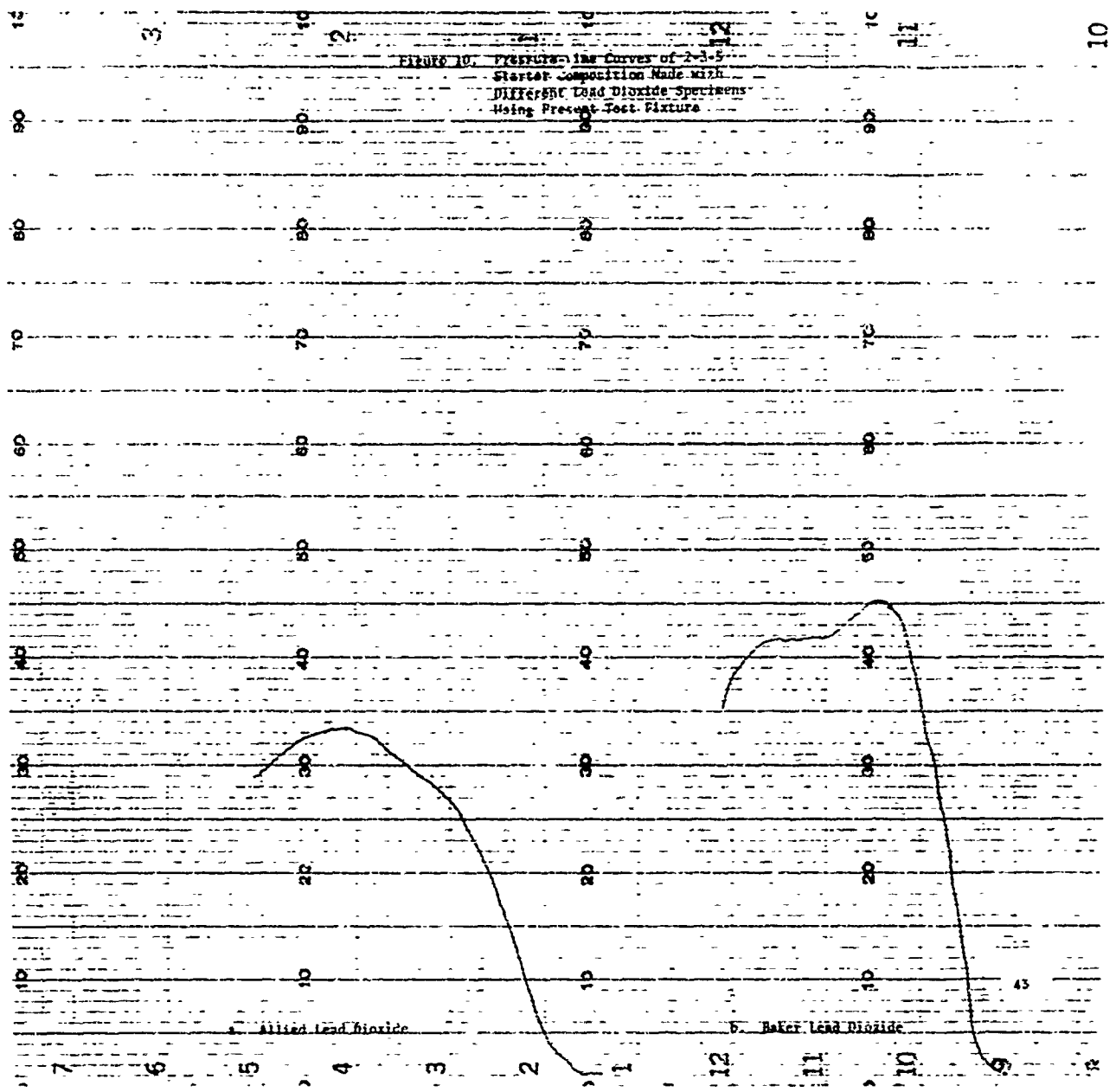


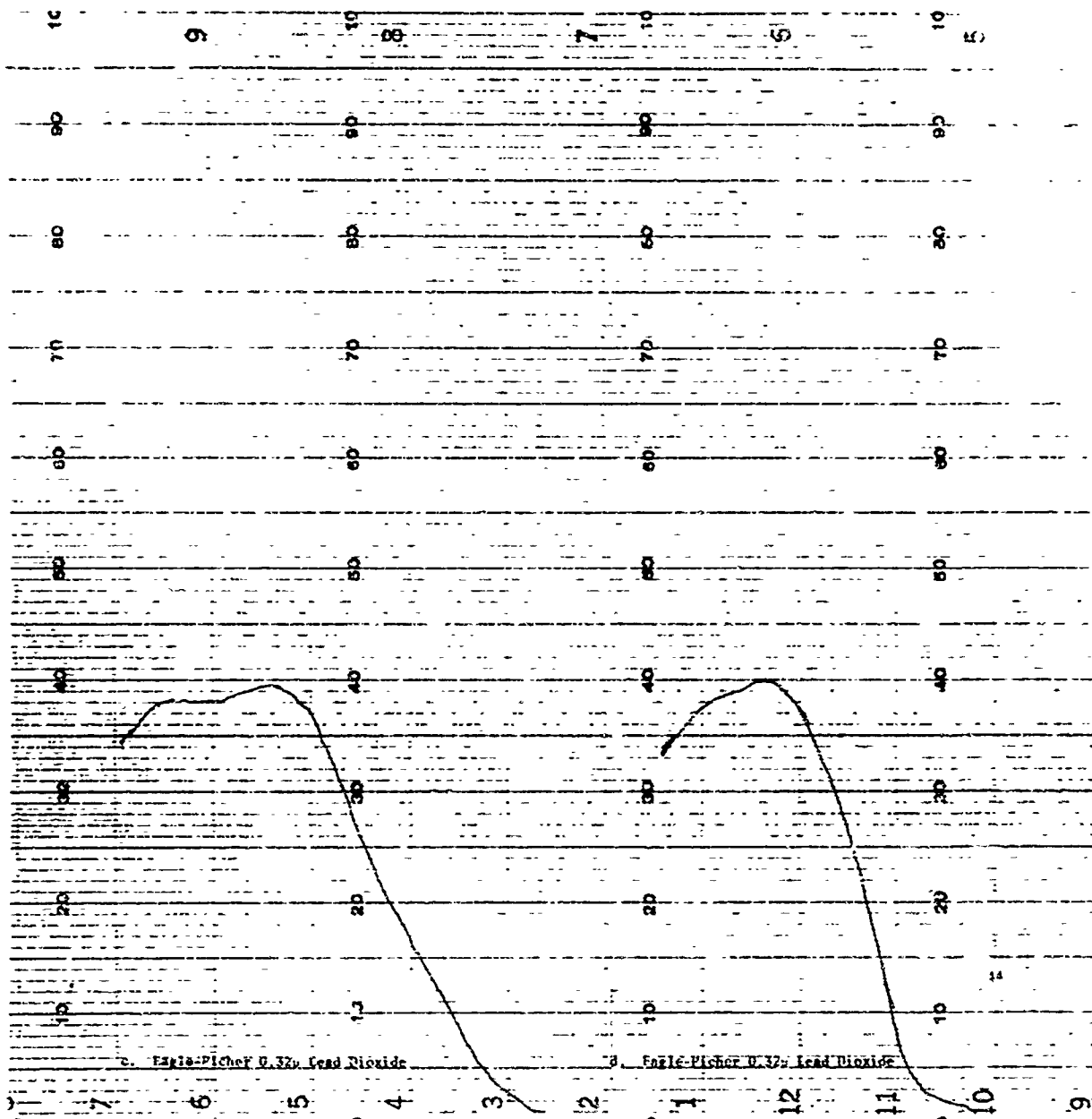
f. Pepcon Lead Dioxide

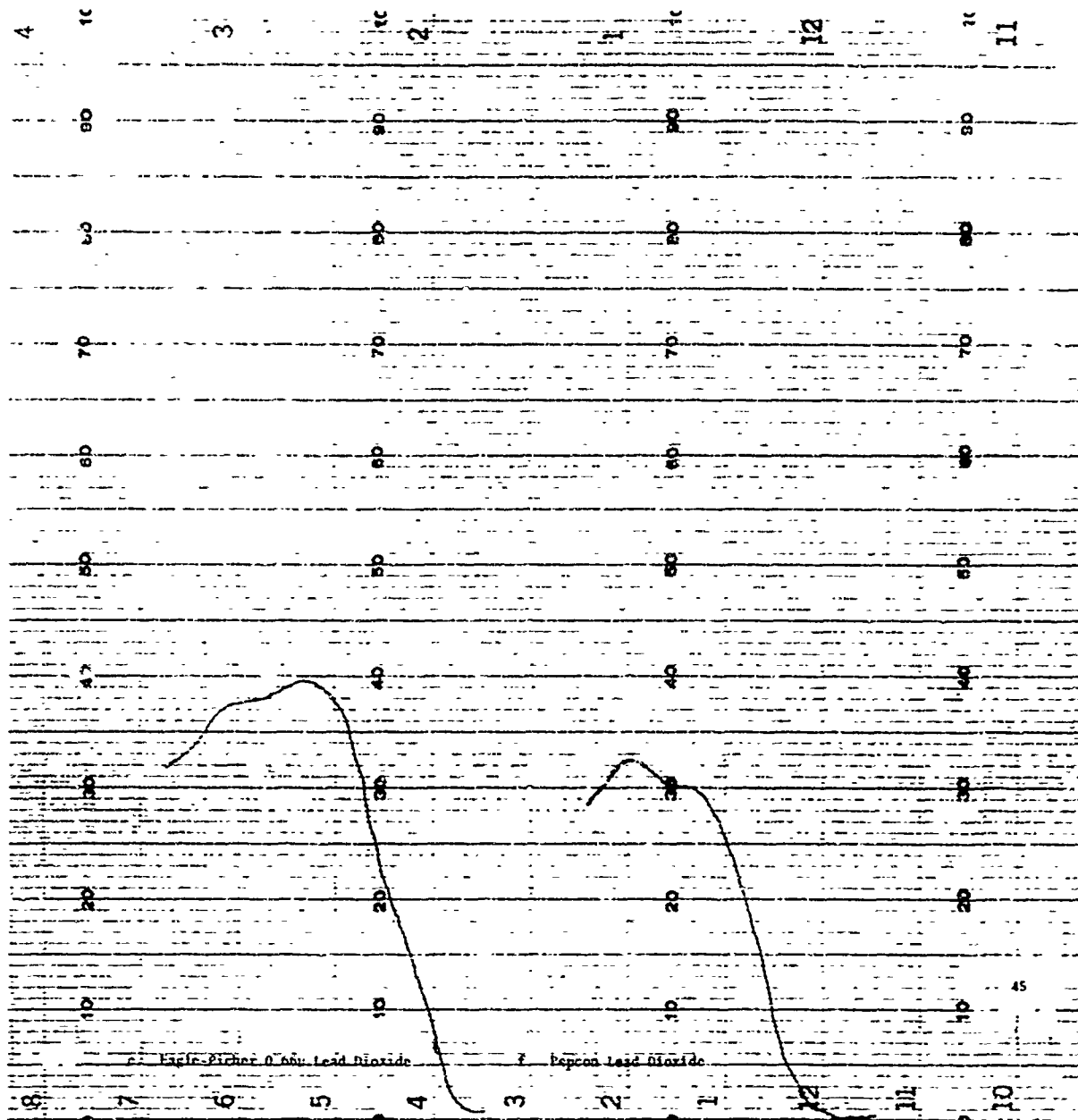


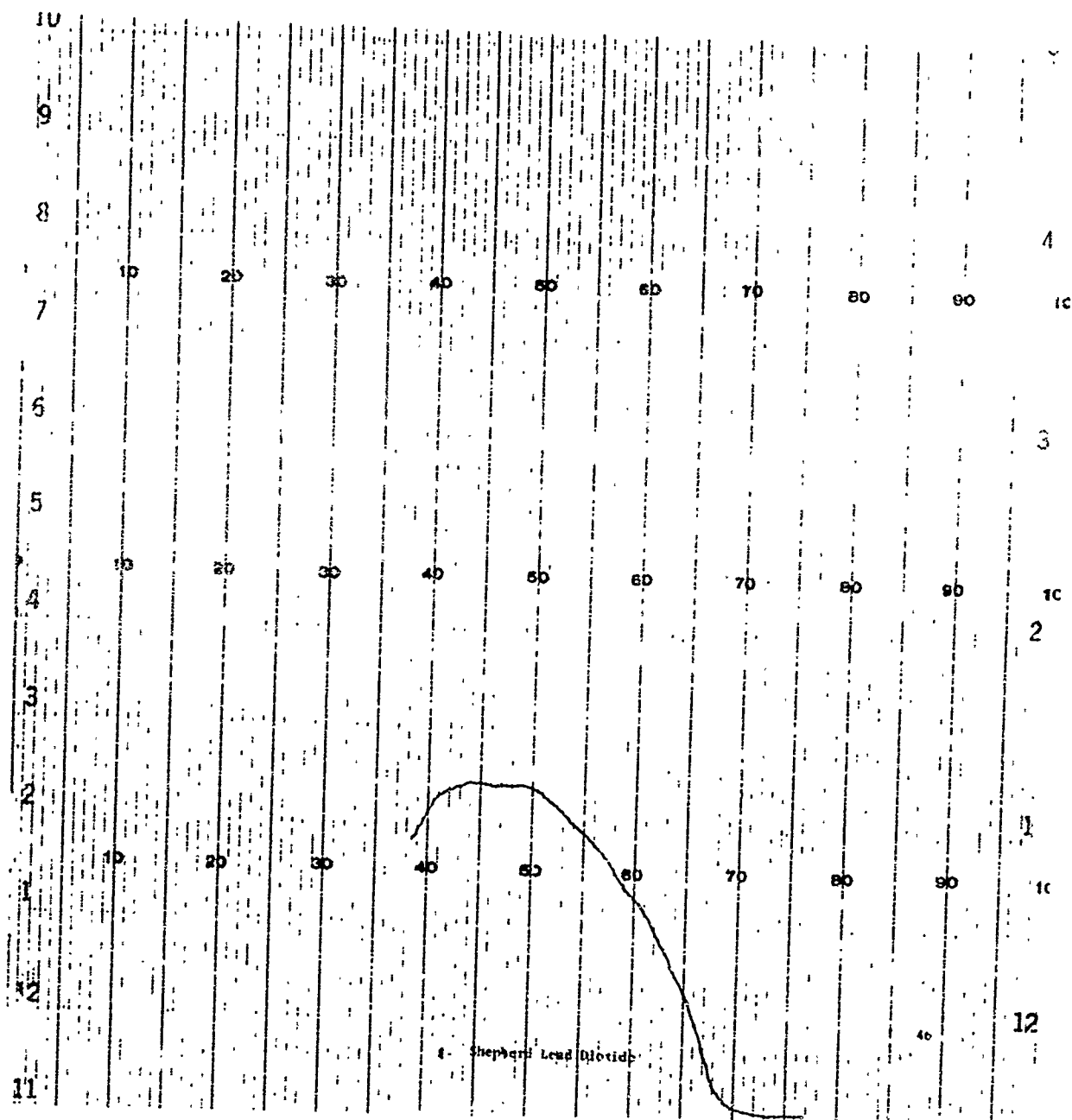
g. Shepherd Lead Dioxide











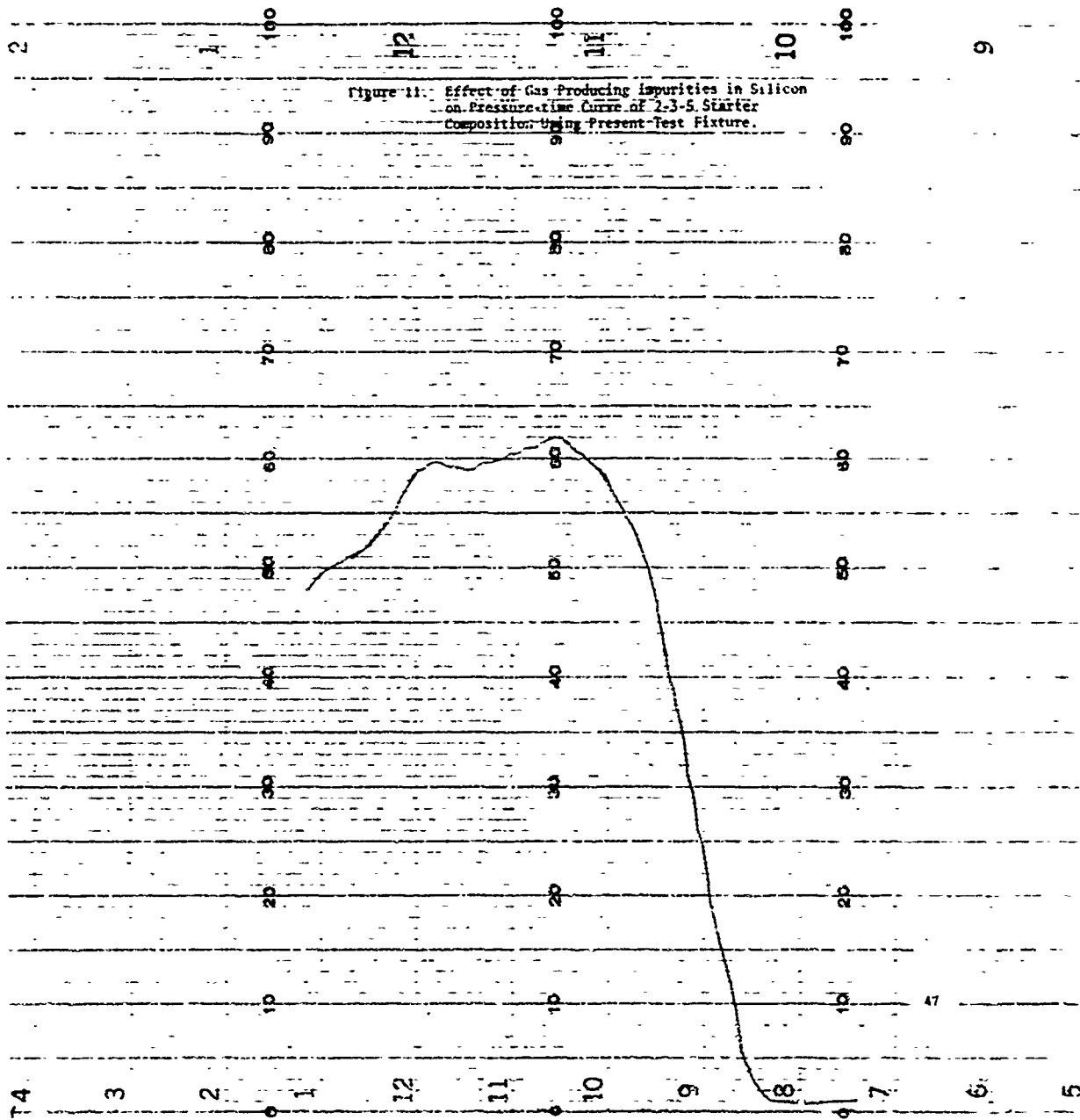
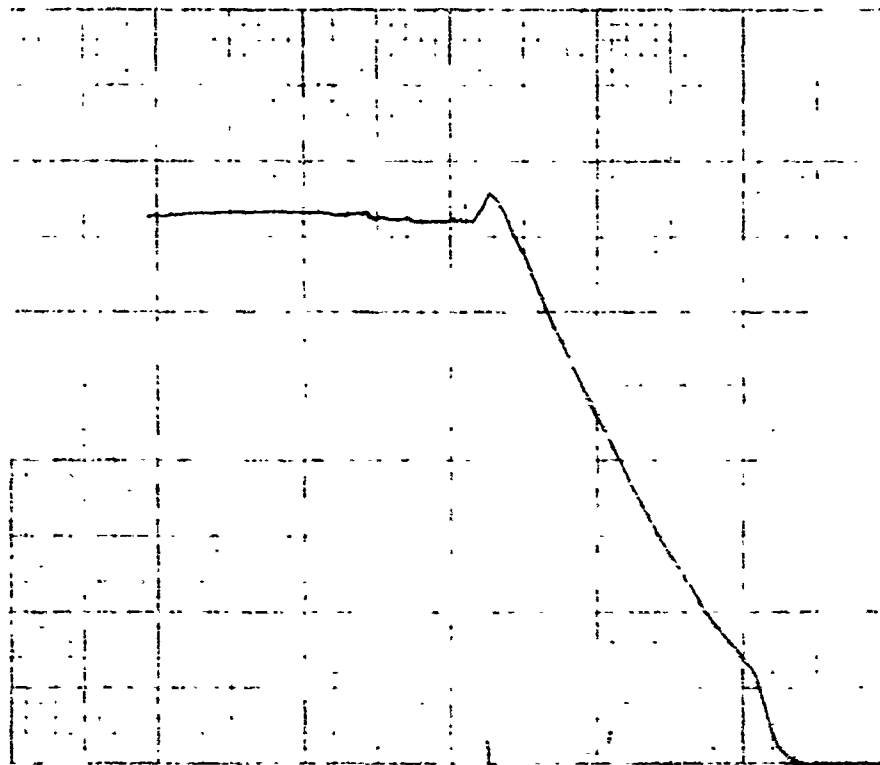


Figure 12. Effect of Gas Producing Impurities in Silicon
on Pressure-time Curve of 2-3-5 Starter
Composition Using New Test Fixture



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| 13 ABSTRACT Difficulties in interpreting pressure-time curve data obtained on the present test fixture (Mk 25 Marine Location Marker Starter Composition) has led to the design of a new test fixture with a sample geometry which produces a linear plot. A comparison of results obtained on the two test fixtures is made. An optimum range of burning characteristics for the new test fixture is recommended. Also recommended is a basic starter composition formula with enough flexibility to compensate for performance variations due to variations in the reactivity of the chemical ingredients. | | |

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| Starter Composition Pressure-Time Curves Pressure-Time Test Fixture Burning Characteristics Mk 25 Marine Location Marker Performance Variations Silicon Lead Dioxide Cupric Oxide Reproducibility of Results William L. Ripley | | | | | | |

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